

Supplementary Information

Metal Nanofoams via a Facile Microwave-assisted Solvothermal Process

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Experimental Section:

Synthesis of Metal Nanofoams: The metal nanofoams of copper, silver, and nickel were produced by dissolving 200 mg of copper(II) acetate monohydrate (Alfa-Aesar), silver-acetate (Fischer Scientific, laboratory grade), or nickel(II) acetate tetrahydrate (Arcos Organics, 99+%) in 12 mL of dry tetraethylene glycol (TEG) (Sigma-Aldrich 99%). The TEG was dried by storing on 4A molecular sieves (Fisher Scientific) prior to use. Dissolution of the salts was expedited with approximately 10 min of ultra-sonication creating translucent blue, green, and clear solutions, respectively, for copper, nickel, and silver. The solutions were subsequently transferred to 20 mL borosilicate vessels and heated as fast as possible with a max power of 850 W to 300 °C in an Anton-Paar Monowave microwave reactor. The solutions were not stirred. Stirring of the solutions would cause the foams to become compacted into a ball of metal. The reactions were stopped approximately 1 min after the observation of the pressure spike. The samples were then washed three times with acetone and dried in an air oven at 100 °C.

Characterization of Metal Nanofoams: X-ray diffraction patterns were collected on a Rigaku Miniflex 600 desktop x-ray diffractometer with copper K α radiation ($\lambda = 1.54184 \text{ \AA}$). The diffraction patterns were collected in continuous mode over a range of $10 - 80^\circ 2\theta$ at a rate of 3° per minute. Subsequently, the average crystallite size was determined with Rietveld refinement on Rigaku PDXL 2 software. The morphology of the samples was investigated with a JEOL JSM-5610 scanning electron microscope (SEM). The samples were prepared by affixing a small foam sample onto a carbon tape on a sample

holder. The surface area of the samples was determined with a Quantachrome autosorb iQ2 Brunauer, Emmett, and Teller (BET) analyzer. Approximately 100 mg of each of the metal nanofoams was placed in a 10 mL sample holder, outgassed at 100 °C overnight, and subjected to a multi-point BET adsorption/desorption test. The surface areas were then calculated with the multi-point BET method. The correlation coefficients for all of the BET surface area fits were greater than 0.999. The transmission electron microscopy (TEM) was carried out with a JEM-2100F microscope with an accelerating voltage of 200 kV. The energy dispersive x-ray spectroscopy (EDS) was performed with the JEM-2100F microscope equipped with an Oxford Instruments EDS detector while operating in the scanning transmission electron microscopy mode.

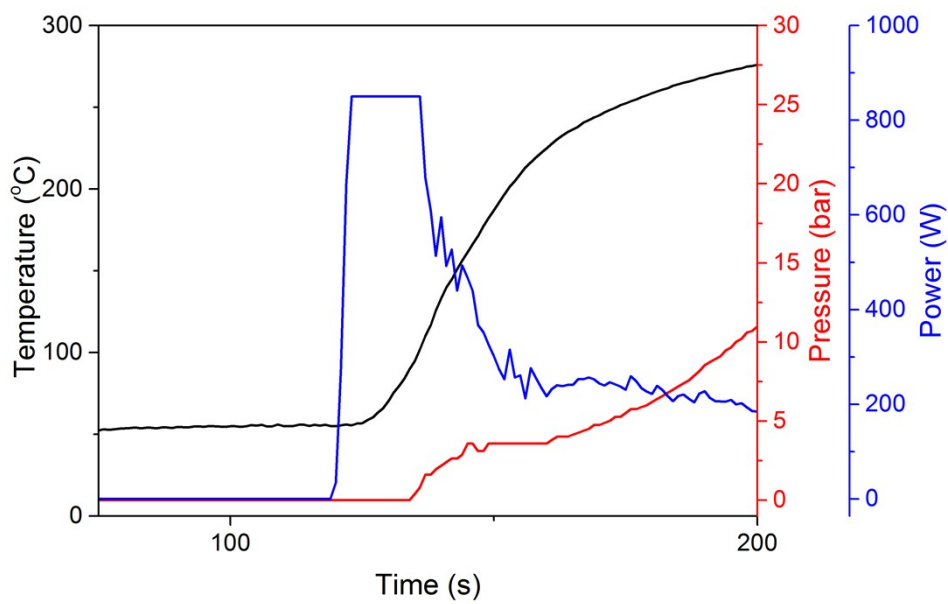


Figure S1 Pressure, temperature, and power versus time of the Anton-Paar microwave reactor during the synthesis of silver metal nanofoam.

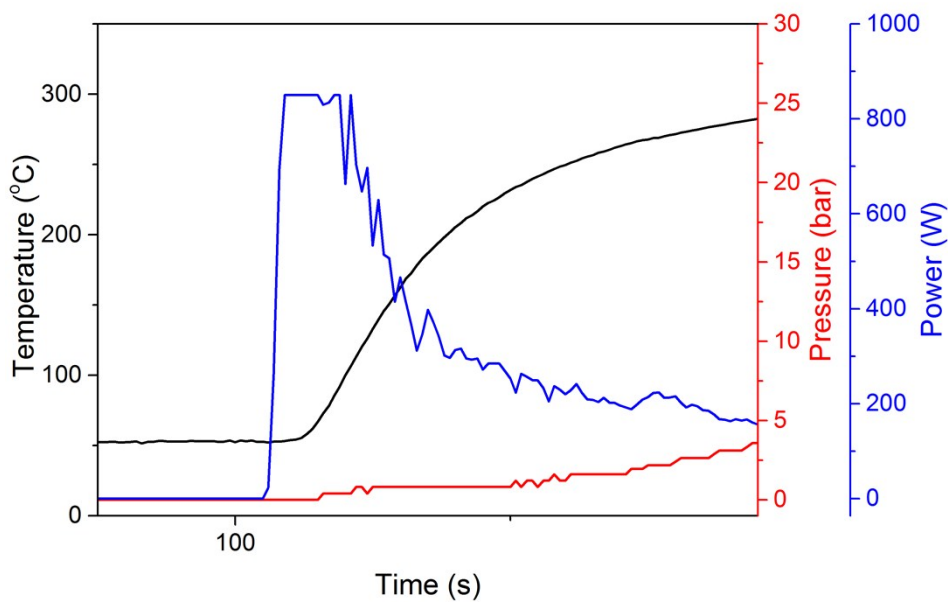


Figure S2 Pressure, temperature, and power versus time of the Anton-Paar microwave reactor during the synthesis of copper metal nanofoam.

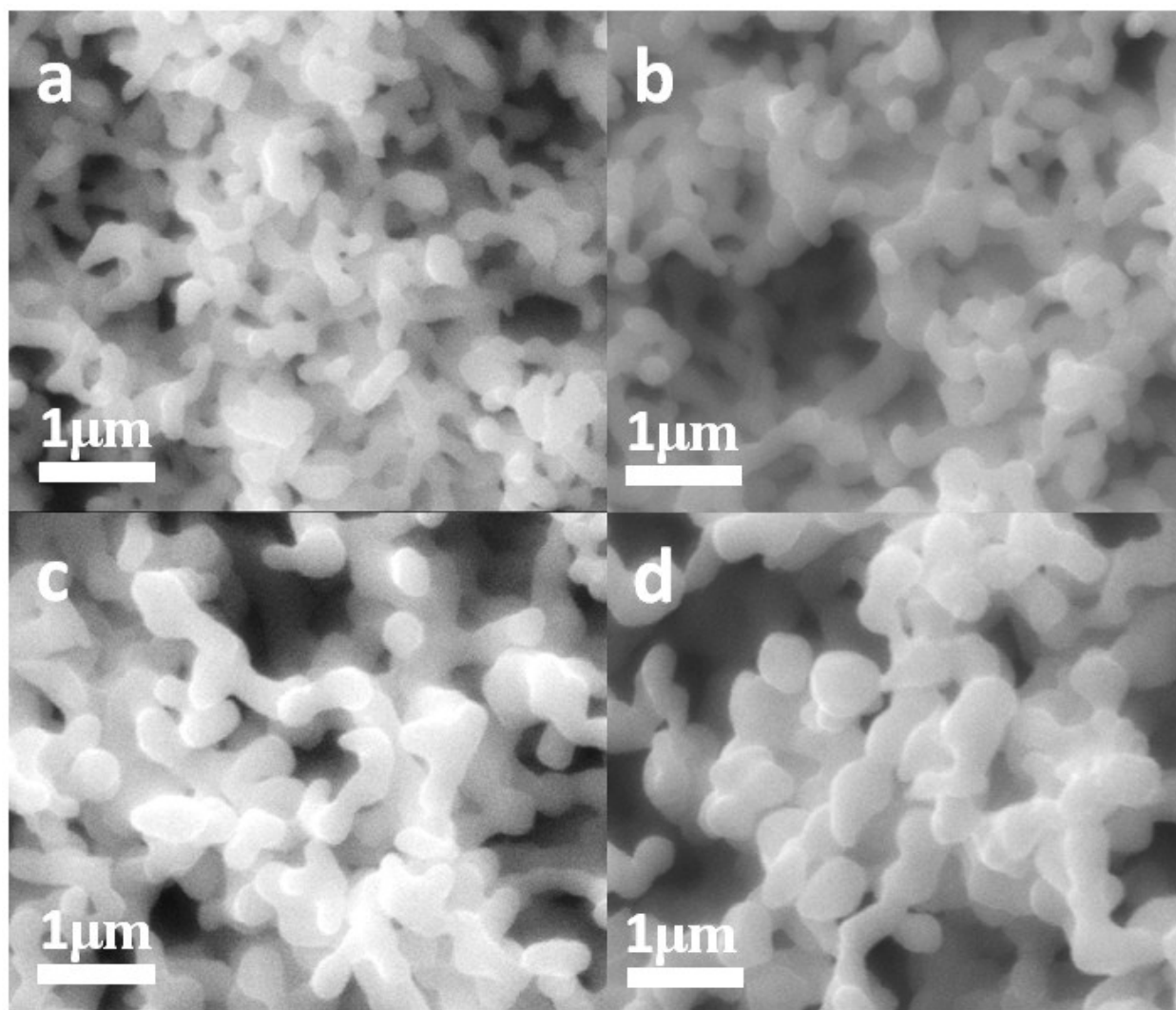


Figure S3 SEM micrographs of the copper foams formed from different concentrations of copper acetate: (a) 0.01 M, (b) 0.02 M, (c) 0.04 M, and (d) 0.06 M.

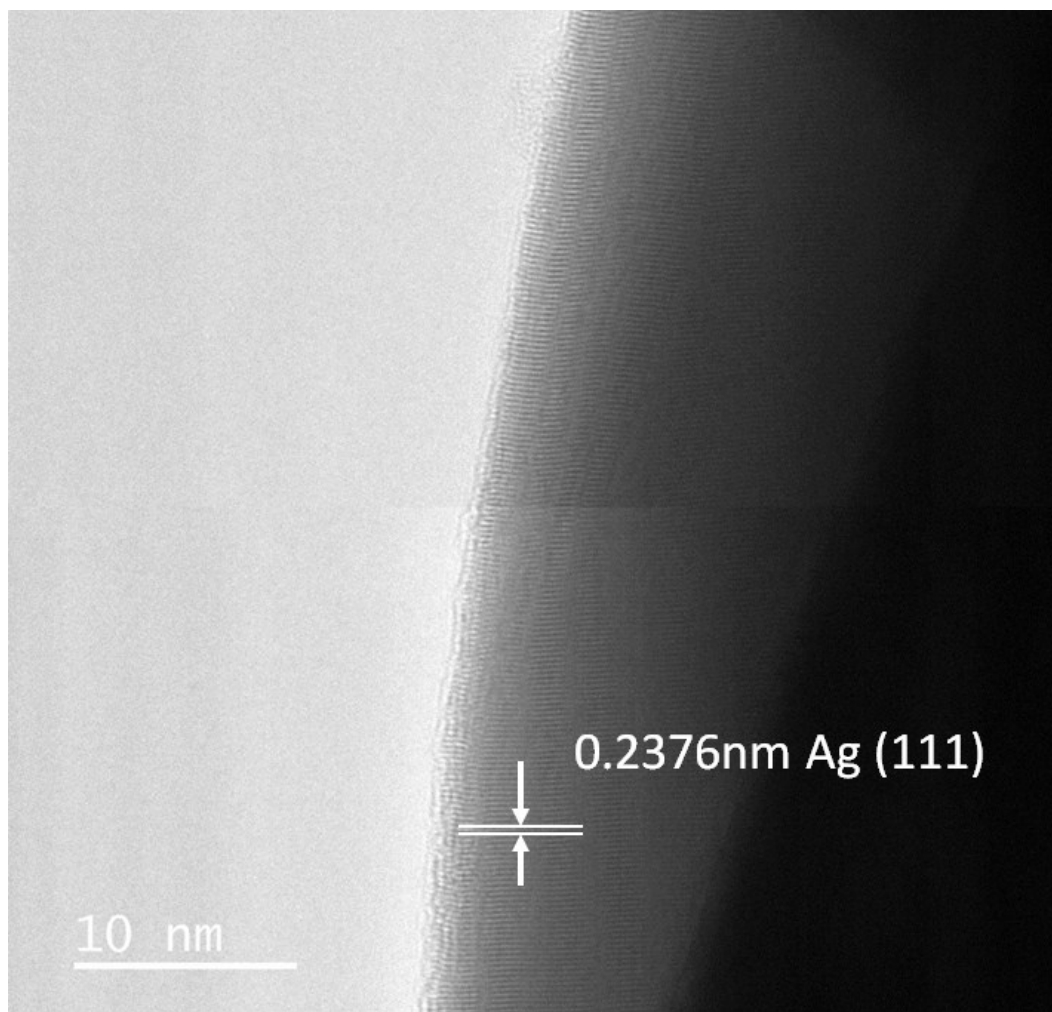


Figure S4 TEM micrograph of the silver foam with interplanar spacing corresponding to the (111) plane.

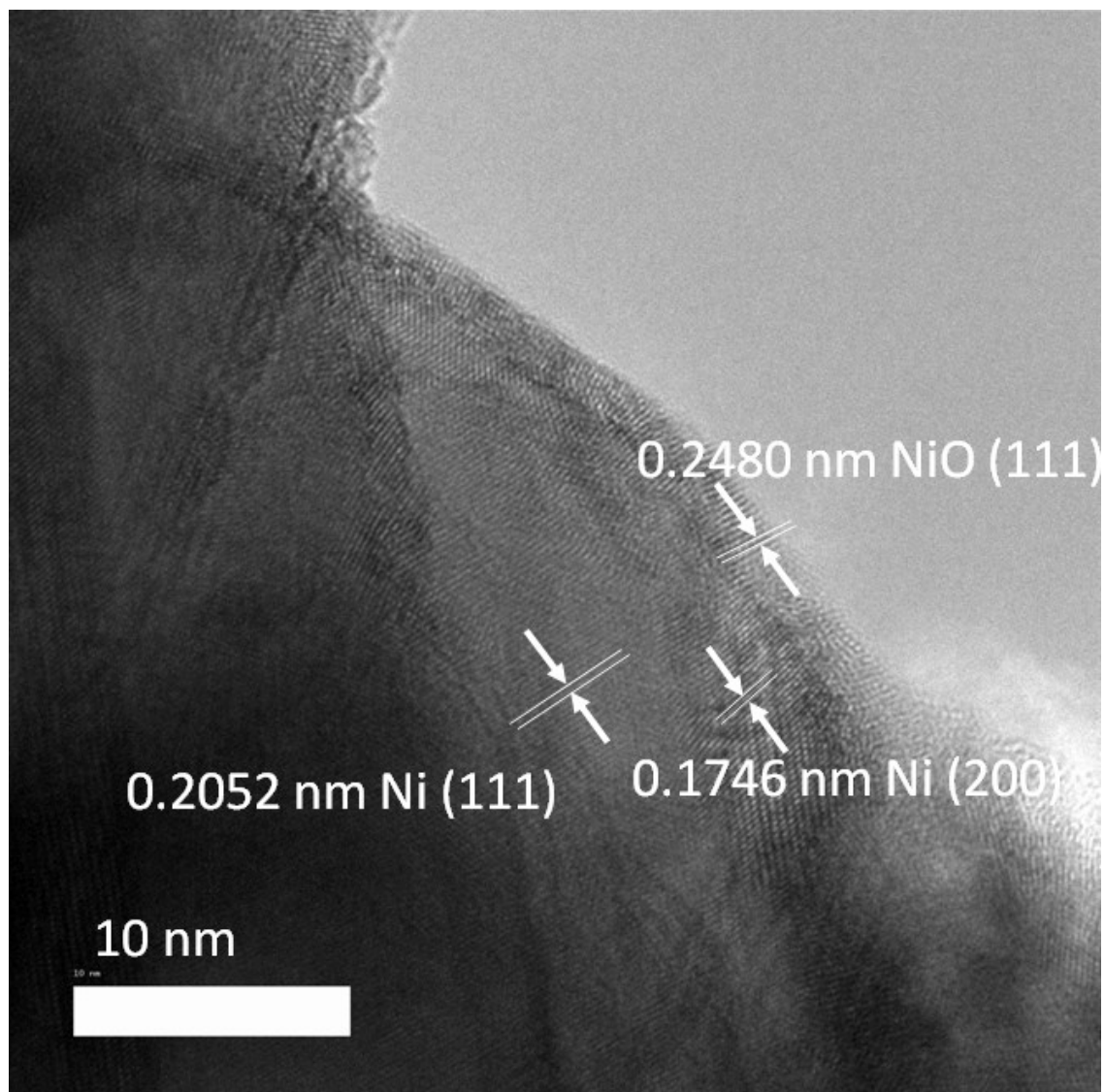


Figure S5 TEM micrograph of the of nickel foam with the interplanar spacings in the bulk corresponding to the Ni (111) and (200) planes and at the surface corresponding to the NiO (111) planes. The micrograph shows that the particle is mostly metallic Ni with a 1 – 3 nm thick layer of NiO on the surface.

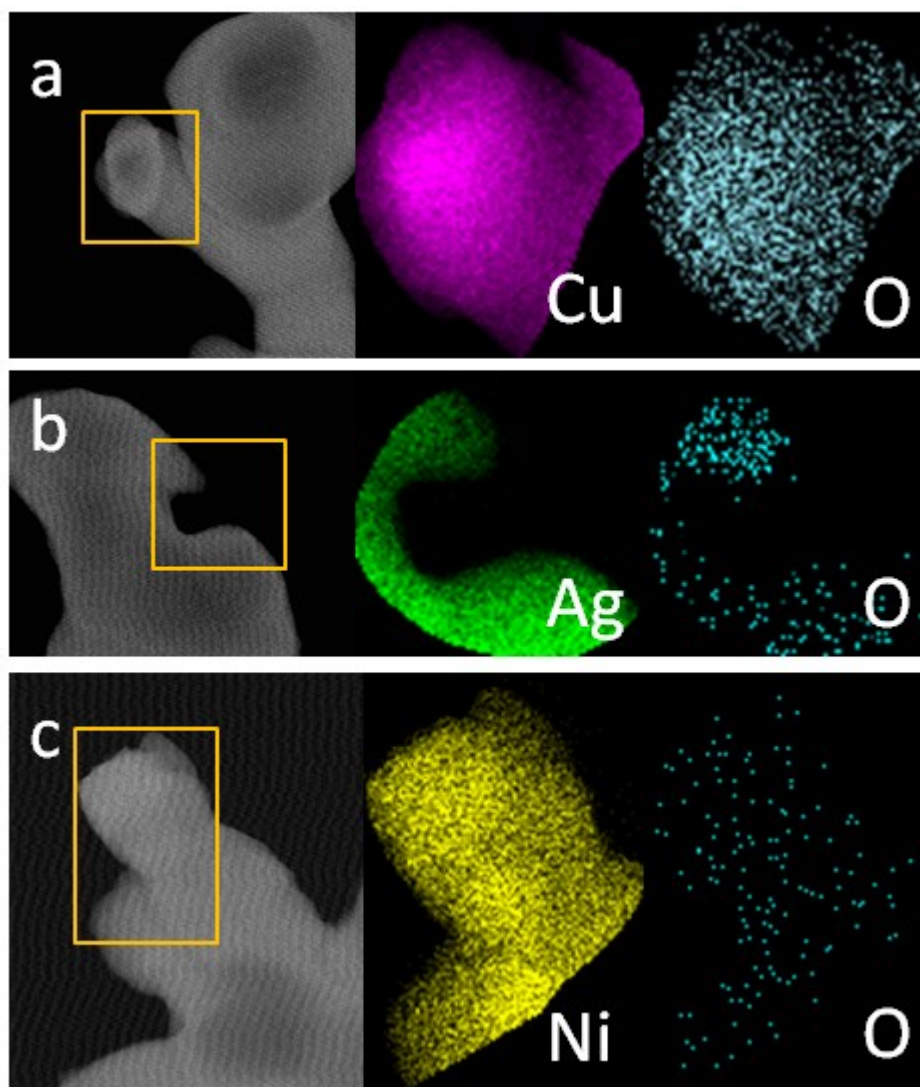


Figure S6 STEM micrographs and EDS maps of the (a) copper, (b) silver, and (c) nickel nanofoams.

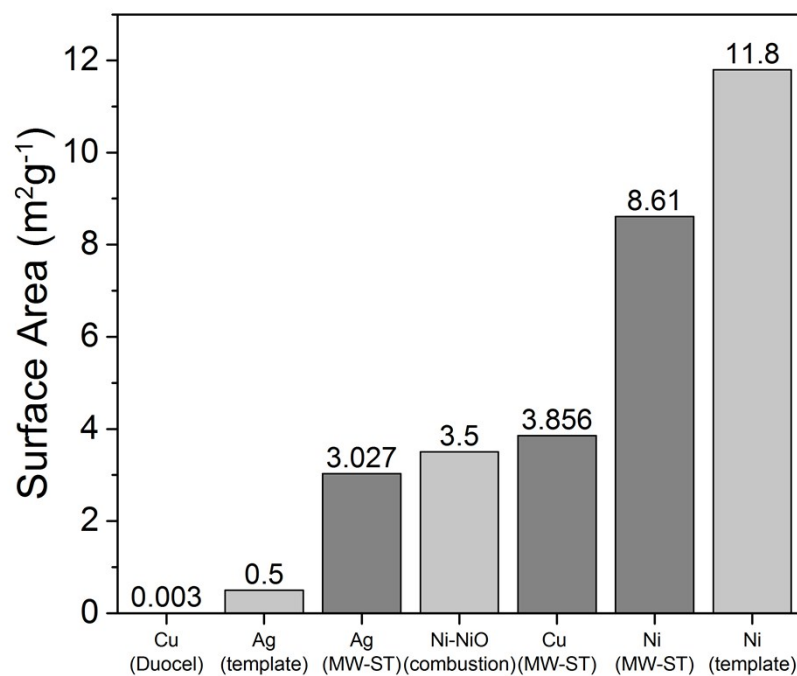


Figure S7 BET surface area of the MW-ST copper, silver, and nickel nanofoams compared to those of commercial copper foam³¹, silver (templated) foam¹⁸, nickel-nickel-oxide (combustion) foam³², and nickel (templated) foam.¹⁹

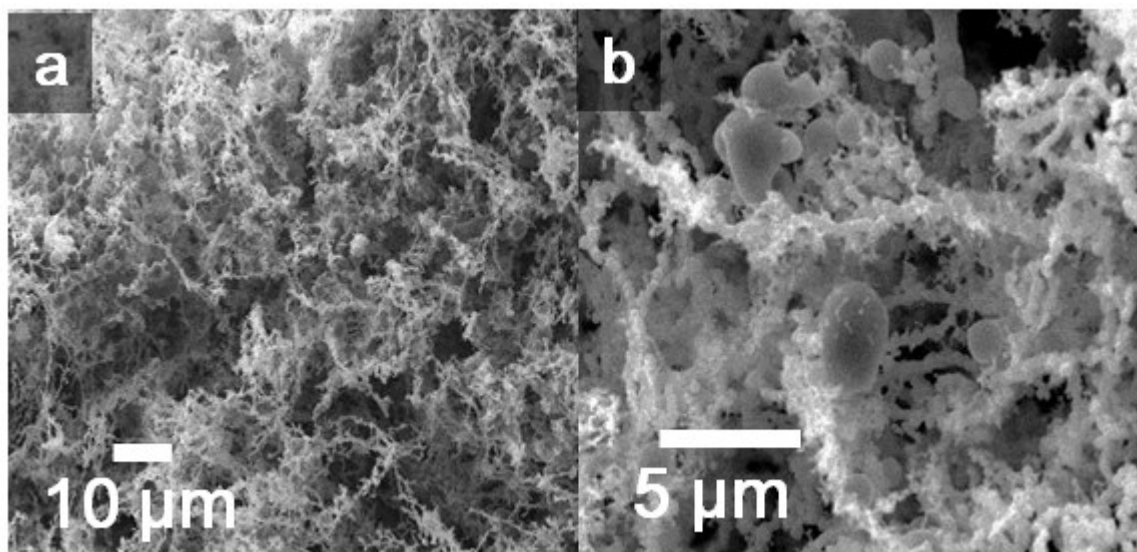


Figure S8 SEM micrographs of the nickel foam after 5 min of additional microwave heating shown at (a) low magnification and (b) high magnification.

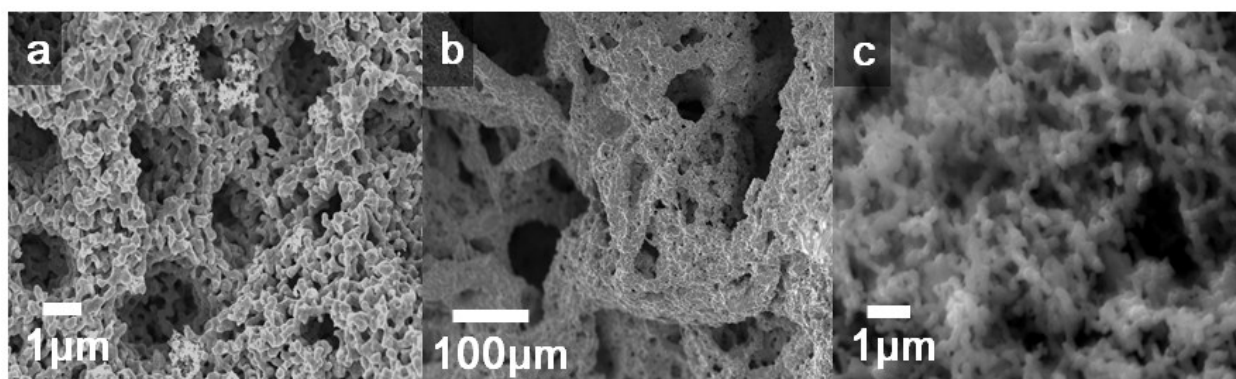


Figure S9 SEM micrographs of the (a) copper, (b) silver, and (c) nickel nanofoams after post heating in an argon atmosphere at 600 °C for 4 h.